B97 A Study of the Thermal Decomposition of Nitrate Ester Explosives by Gas Chromatography/Vacuum Ultraviolet Spectroscopy (GC/VUV) and Its Application to Post-Blast Debris

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Learning Overview: After attending this presentation, attendees will be better informed about the thermal decomposition of nitrate ester explosives and its impact on analyzing realistic post-blast debris.

Impact on the Forensic Science Community: This presentation will impact the forensic science community by discussing a recently developed detector for GC/VUV and its applicability to the analysis of nitrate ester explosives and post-blast debris.

The method employed in this research consisted of an Agilent® 7890B GC (50°C hold 0.5min, ramp 20°C/min to 280°C) with a multimode inlet (50°C to 280°C at 900°C/min), a hydrogen carrier gas (3.2ml/min flow rate), and a Restek® Rtx-5MS column (15m x 0.32mmID x 0.25um) was utilized in line with a VGA-101 VUV spectrometer. The VUV spectrometer was operated with nitrogen as the make-up gas (0.35psi) and a transfer line/flow cell temperature range of 190°C to 300°C between 125nm and 430nm. This method was utilized to analyze the thermal decomposition of four nitrate ester explosives: NG, EGDN, ETN, and PETN, as well as post-blast debris of double-base smokeless powders.

Nitrate ester explosives thermally decompose in the transfer line of the VUV detector into nitric oxide, carbon monoxide, formaldehyde, oxygen, and water. The explosives decompose into differing stoichiometric ratios of the decomposition products and contribute to variation in each nitrate ester explosive VUV spectrum. Due to adherence to Beer Lambert Law, the percentage of each decomposition product was determined by spectral subtraction. The percent contribution of the decomposition products at 300°C was optimized using the Excel® Solver tool. The explosives decreased in concentration with increasing temperature in a logistical fashion. Using JMP software, a 2 parameter logistic fit was utilized to identify the inflection point (the point at which 50% of explosives were degraded) of the sigmoidal curve. These temperatures ranged from 243.74°C to 248.23°C.

The sensitivity of the GC/VUV for nitrate ester explosives was evaluated by looking at the peak area over the range of transfer line/flow cell temperatures. Results indicated that at lower temperatures, NG and ETN had greater sensitivity, while PETN had variable responses and EGDN did not have significant changes in peak area with changing temperatures. Limits of detection for these compounds analyzed with a transfer line/flow cell temperature of 240°C were calculated at low ppm.

To investigate the applicability to forensic post-blast debris analysis, the Indiana State Police Bomb Squad assisted in obtaining realistic samples of post-blast residues of double-base smokeless powder (Alliant Red Dot) on galvanized steel and Polyvinyl Chloride (PVC) pipe fragments. The pipes were placed in a perforated steel box prior to initiation to allow for collection of the post-blast debris for analysis. These samples were analyzed using the same method described above at a transfer line/flow cell temperature of 240°C. The analysis of the post-blast debris of these two types of devices showed that, for the first time, GC/VUV can be used to identify NG as the energetic material with diphenylamine and ethyl centralite as additives in post-blast debris.

Gas Chromatography, Vacuum Ultraviolet Spectroscopy, Explosive Analysis

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